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मानक

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Mazdoor Kisan Shakti Sangathan

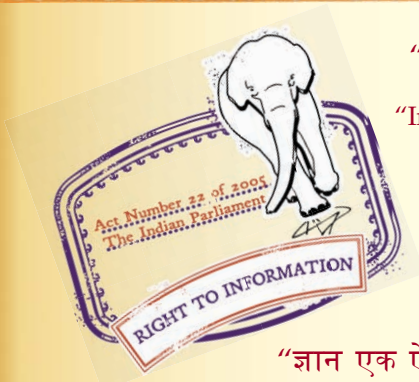
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“Step Out From the Old to the New”

IS 12210 (1987): Caprolactam [PCD 12: Plastics]



“ज्ञान से एक नये भारत का निर्माण”

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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 12210 - 1987

Indian Standard

SPECIFICATION FOR CAPROLACTAM

UDC 547.473.2/.3

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

Gr 7

September 1988

**AMENDMENT NO. 3 JUNE 2008
TO
IS 12210 : 1987 SPECIFICATION FOR
CAPROLACTAM**

(Page 5, Table 1) — Delete ‘*’ mark at Sl No. 5, col 3 and the subsequent footnote with * mark.

(PCD 9)

Reprography Unit, BIS, New Delhi, India

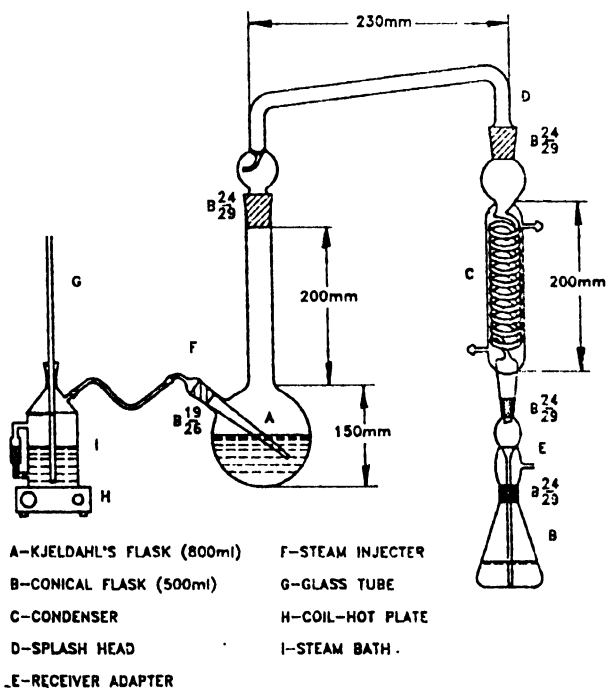
AMENDMENT NO. 2 APRIL 1995
TO
IS 12210 : 1987 SPECIFICATION FOR CAPROLACTAM

[Page 5, Table 1, Sl No.(iii), col 3] — Substitute '69.0' for '68.8'.

[Page 5, Table 1, Sl No.(x), col 2 and 3] — Substitute 'Transmittance' for 'Absorbance' and '92 Min.' for '0.05 Max'.

(Page 10, clause A-3.3, lines 3 and 4) — Substitute '500-ml conical flask' for '250-ml conical flask'.

(Page 11, Fig. 2) — Substitute the following for the existing figure:



**FIG. 2 DISTILLATION APPARATUS FOR DETERMINING
 VOLATILE BASES IN CAPROLACTUM**

Amend No. 2 to IS 12210 : 1987

(*Page 18, clause A-7.1, last line*) — Substitute 'and 10 mm cell' for 'and 10 nm cell'.

(*Page 18, clause A-7.1.1, second equation*) — Substitute ' $2I_2$ ' for ' I_2 '.

(*Page 20, clause A-7.5, line 3*) — Substitute '... lactam with a small content of cyclohexanone oxime' for '... lactam with a small amount of cyclohexanone oxime'.

(*Pages 25 and 26, clauses A-11, A-11.0, A-11.1 and A-11.4*) — Substitute 'transmittance' for 'absorbance'.

(*Page 26, clause A-11.3.1, line 8*) — Substitute '100 transmittance' for 'zero absorbance'.

(*Page 26, clause 11.3.2, lines 4 and 5*) — Substitute the following for the existing:

'read 100 since it has been used to adjust the instrument to 100 transmittance.'

AMENDMENT NO. 1 FEBRUARY 1995
TO
IS 12210 : 1987 SPECIFICATION FOR CAPROLACTUM

(*Page 4, clause 0.6, line 5*) } — Substitute '290 nm' for '290 mm'.
[*Page 5, Table 1, Sl No. (x)*] }
(*Page 7, clause A-2.2.3, lines 1 and 2*) — Substitute 'less than' for 'less
then' at both the places.

(PCD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR CAPROLACTAM

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(Continued on page 2)

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IS : 12210 - 1987

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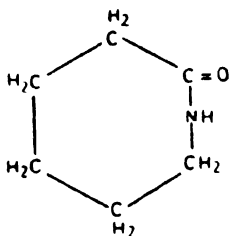
Indian Standard

SPECIFICATION FOR CAPROLACTAM

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 1 September 1987, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Caprolactam [$\text{CH}_2(\text{CH}_2)_4\text{NHCO}$] is used in the manufacture of synthetic fibres, plastics, bristles, film, coatings, synthetic leather, plasticizers and paint vehicles. It is also used as cross-linking agent for curing polyurethane. It is represented by the following structural formula:



0.3 Oxidizable impurities in caprolactam constitute a critical quality parameter. From a study of literature, it transpired that this characteristic has been variously described as permanganate number, permanganate index, permanganate consumption or permanganate absorbance. The Committee, after due consideration, decided to adopt permanganate number for describing this parameter (*see A-2*). For determining permanganate number, two methods, using 1 and 3 percent aqueous solution of caprolactam, were found to be in vogue. However, the Committee, after due consideration, decided to adopt determination of permanganate number using 1 percent aqueous solution for the present. In due course, after adequate data is generated using 1 and 3 percent aqueous solution, the Committee may review the test method on its own merits taking cognizance of the precision of the method.

0.4 Till recently, inconsistent supply of desired quality of benzene, an important raw material for the manufacture of caprolactam, was considered to be a major hurdle to cater to the needs of synthetic fibre and other user industries. However, in view of the subsequent improvement

at the refineries, the relevant standards for benzene are currently being revised in order to modify the same suitably to ensure indigenous production of caprolactam of acceptable quality.

0.5 The requirement of iron content has been stipulated on the basis of data available from the only manufacturer so far in the country, Gujarat State Fertilizers Co Ltd, Vadodara. This requirement may be reviewed at a later date when other prospective manufacturer, Fertilizers and Chemicals Travancore Ltd, Udyogamandal, Cochin goes into full-fledged commercial production and adequate additional data is made available.

0.6 Considerable assistance has been derived, in the preparation of this standard, from the overseas specifications and the following documents issued by the International Organization for Standardization (ISO):

ISO 7059-1982	Caprolactam for industrial use --- Determination of absorbance at a wavelength of 290 nm
ISO 7060-1982	Caprolactam for industrial use --- Determination of crystallizing point
ISO 8112-1984	Caprolactam for industrial use - Determination of colour of 50 percent aqueous caprolactam solution. expressed in Hazen units (Platinum-cobalt scale) --- Spectrometric method
ISO/DIS 8660	Caprolactam for industrial use --- Determination of permanganate index --- Spectrometric method
ISO/DIS 8661	Caprolactam for industrial use --- Determination of volatile bases content -- Titrimetric method after distillation

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for caprolactam.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of crystals or flakes.

2.2 The material shall also comply with the requirements given in Table 1

*Rules for rounding off numerical values (*revised*).

when tested according to the methods prescribed in Appendix A. References to the relevant clauses of Appendix A are given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR CAPROLACTAM
(Clause 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Permanganate number (1 percent aqueous solution), seconds, <i>Min</i>	10 000	A-2
ii)	Volatile base (as NH_3), meq/kg, <i>Max</i>	0.5	A-3
iii)	Solidification point, °C, <i>Min</i>	68.8	A-4
iv)	Colour (50 percent aqueous solution), APHA, <i>Max</i>	5	A-5
v)	Iron content, ppm, <i>Max</i>	0.1*	A-6
vi)	Oxime content, ppm, <i>Max</i>	10	A-7
vii)	Free acidity, meq/kg, <i>Max</i> <i>or</i> Free alkalinity, meq/kg, <i>Max</i>	0.05 } 0.1 }	A-8
viii)	Moisture content, percent by mass, <i>Max</i>	0.1	A-9
ix)	Matter insoluble in water, ppm, <i>Max</i>	10	A-10
x)	Absorbance (50 percent aqueous solution) in 1 cm cell at 290 nm, <i>Max</i>	0.05	A-11

*This is subject to review as and when FACT plant goes into commercial production.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in either 25 kg bags with LDPE liner and covered with paper bags or as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and marked legibly and indelibly with the following information:

- Name of the material;
- Name of the material in the container;
- Name of the manufacturer and his recognized trade-mark, if any; and
- Lot or batch number in code or otherwise together with date of manufacture.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative test sample of the material and the criteria for conformity shall be as prescribed in Appendix B.

A P P E N D I X A

(Clause 2.2, Table 1)

METHODS OF TEST

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF PERMANGANATE NUMBER

A-2.0 General — Permanganate number is a measure of oxidizable impurities present in caprolactam. For this standard, it is measured as the time taken by 1 percent aqueous solution of caprolactam with 0.01 N potassium permanganate solution to match in colour with that of standard reference solution of copper sulphate-cobaltous chloride solution measured in seconds.

*Specification for water for general laboratory use (*second revision*).

A-2.1 Apparatus

A-2.1.1 Colour Comparison Tube (Matched) — Nessler tubes with marks at 50 and 100 ml. provided with ground-on, optically clear glass caps.

A-2.1.2 Constant Temperature Bath — Capable of maintaining a temperature of $20.0 \pm 0.5^\circ\text{C}$. It is important that the constant temperature bath be protected from direct light. If a glass constant temperature bath is employed, it should be wrapped or coated with an opaque material.

A-2.1.3 Pipette — Capable of delivering 1.0 ml of solution.

A-2.1.4 Interval Timer and Clock — Capable of measuring a time interval of 120 minutes or more. An alarm arrangement is desirable.

A-2.2 Reagents

A-2.2.1 Potassium Permanganate Solution (0.01 N) — Dissolve 0.316 g of the potassium permanganate and dilute to 1 litre with freshly boiled and cooled water. Clean glassware is absolutely essential for the stability of this solution. The solution should be stored in brown bottle and be prepared freshly every day.

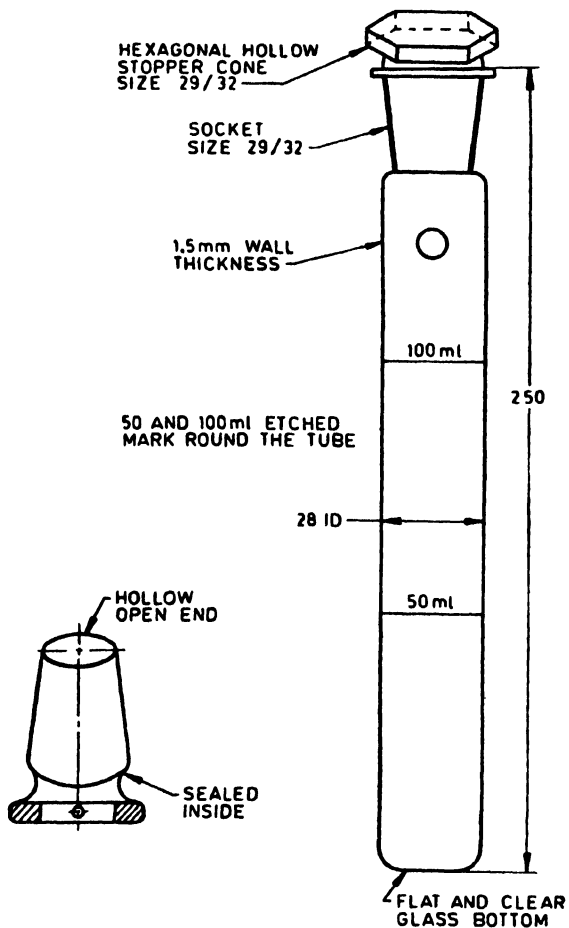
A-2.2.2 Copper Sulphate-Cobaltous Chloride Standard Solution — Weigh 3 ± 0.001 g cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) and 2 ± 0.001 g copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and dissolve in about 200 ml of water. Transfer to 1 000 ml volumetric flask, dilute to the mark, and mix thoroughly.

A-2.2.3 Double Distilled Water — of pH 6.8 ± 0.1 , conductivity less than $0.5 \mu\text{ mhos/cm}$ and silica less than 1 ppm.

A-2.3 Procedure

A-2.3.1 Weigh accurately 1 ± 0.001 g of caprolactam and transfer it to one Nessler tube, dissolve it and make it to 100 ml with double distilled water (A-2.2.3) and place in the constant temperature bath (20°C). Maintain the water level in the bath approximately 2.5 cm below the top of the tube. When the sample has reached the specified temperature (20°C), add 1 ml of potassium permanganate solution (A-2.2.1). Stopper the tube, invert once to mix the contents, return to the bath and simultaneously start the interval timer and clock.

A-2.3.2 In another Nessler cylinder, fill the standard solution (A-2.2.2) to the 100 ml mark, which serves as a comparison standard. Place this reference Nessler cylinder also in thermostat bath at the same temperature (20°C).



All dimensions in millimetres.

FIG. 1 COLOUR COMPARISON TUBE

A-2.3.3 Measure the time in terms of seconds when the colour of the samples solution matches to that of the reference solution. Stop the interval timer and clock. Note the time in seconds. If the colour of sample solution matches to that with standard reference solution in less than 10 000 seconds, then report the actual seconds as permanganate

number of 1 percent caprolactam aqueous solution at 20°C. If the colour match is not yet reached after 10 000 seconds, the test may be interrupted and report the permanganate number greater than 10 000 seconds (see Fig. 1).

NOTE 1 — Clean sample cylinders, and permanganate storage and handling equipment with concentrated hydrochloric acid (relative density 1.19) to remove residual manganese oxide.

NOTE 2 — Remove the cylinders (when both the colours are matching within 10 000 seconds or requires more than 10 000 seconds and compare it to the colour standard by viewing downward through the tubes against a white background from which diffused white light is reflected.

A-3. DETERMINATION OF VOLATILE BASES

A-3.0 General — In the production of caprolactam, basic products (ammonia and organic bases) of volatile nature are formed, which have an unfavourable effect upon the degree of polycondensation as well as upon the process of spinning and stretching.

A-3.0.1 Principle — In a standard high efficiency apparatus, a known amount of sample is treated with alkali, and the volatile bases expelled are fixed up in standard sulphuric acid. The excess acid is determined by titration with standard alkali.

A-3.1 Apparatus

A-3.1.1 Distillation Apparatus — see Fig. 2.

A-3.1.2 Wash-bottle

A-3.1.3 Conical Flasks — 500-ml.

A-3.1.4 Measuring Cylinders — 250-ml.

A-3.1.5 Microburette — 10-ml.

A-3.2 Reagents

A-3.2.1 Sodium Hydroxide Solution — 0.01 N and 30 percent.

A-3.2.2 Sulphuric Acid — 0.01 N.

A-3.2.3 Tashiro-Indicator — Measure 28 ml of methyl red (0.1 percent of the Na-salt in distilled water) and 7 ml of methylene blue (0.1 percent in distilled water) to a 100-ml measuring flask; make up to the mark with distilled water and mix.

A-3.2.4 Pumice Grains

A-3.3 Procedure — Weigh about 50 g of the caprolactam sample to the nearest 0.01 g in the Kjeldahl distillation flask (*A*) and add a few pumice grains. Transfer 25 ml of sulphuric acid solution in a 250-ml conical flask (*B*). Add 8 drops of Tashiro indicator and dip the condenser tube inside the acidic solution. Connect the assembly and then add 40 ml of 30 percent caustic soda solution to flask (*A*) through the funnel. The funnel should be washed with distilled water after all the sodium hydroxide solution is transferred to the distilling flask (*A*). Start distillation by heating or by steam at a rate of 100 ml in 30 minutes. Collect about 200 ml distillate. Disconnect the apparatus, wash the condenser with neutral water and collect the washing in the same flask (*B*). The quantity of neutral water used for cleaning condenser and blank should be the same. Titrate the content of the flask (*B*) for the excess of acid with 0.01 N NaOH solution from microburette to colour change from purple to green (V_2 ml). Every time carry out a blank determination without caprolactam to determine the content of volatile bases in same volume of caustic soda solution (40 ml of 30 percent NaOH solution) in the same manner.

A-3.4 Calculation

$$\text{Volatile base as } \text{NH}_3 \text{ (meq/kg)} = \frac{(V_1 - V_2) \times N \times 1000}{M}$$

where

V_1 = volume in ml of 0.01 N NaOH solution used for the blank;

V_2 = volume in ml of 0.01 N NaOH solution used for the sample;

N = normality of 0.01 N NaOH solution; and

M = mass in g of the sample taken.

Volatile base on NH_3 (mg/kg) or ppm

$$= \frac{(V_1 - V_2) \times F \times 0.1703 \times 1000}{M}$$

where

V_1 = volume of 0.01 N NaOH solution used for the blank,

V_2 = volume of 0.01 N NaOH solution used for the sample, and

F = factor of 0.01 N NaOH solution.

NOTE — Amount of NH_3 corresponding to 1 ml of 0.01 N NaOH solution (mg) = 0.1703.

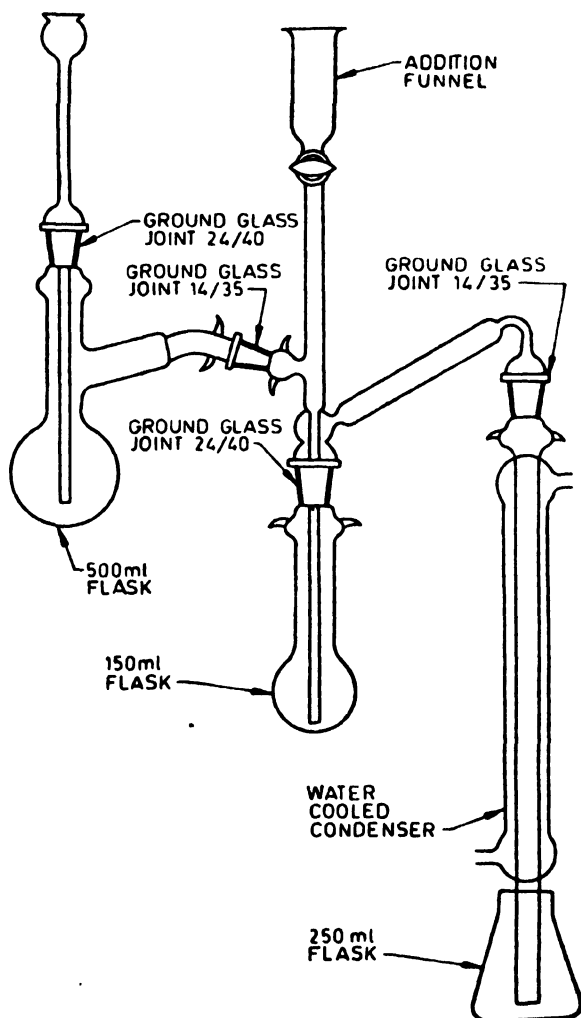


FIG. 2 DISTILLATION APPARATUS FOR DETERMINATION OF VOLATILE BASE IN CAPROLACTAM

A-3.4.1 Report — Report the result as volatile base in terms of meq/kg or volatile base in terms of ammonia mg/kg to the nearest 0.02 meq/kg or 0.34 mg/kg of ammonia.

NOTE — This method is also applicable to aqueous solution of the sample. The amount of the solution to be taken for analysis should contain about 20 g of caprolactam. To transfer the solution quantitatively use about 75 ml of water, add 40 ml of 30 percent sodium hydroxide solution through the funnel.

A-4. DETERMINATION OF SOLIDIFICATION POINT

A-4.0 Principle — A specimen of molten material in a test tube fitted with a thermometer is placed in an air-bath which, in turn, is surrounded by a water-bath held at 60 to 65°C. As the molten material cools, periodic readings of its temperature are taken. When solidification of the material occurs and a minimum rate of temperature change (cooling curve) is observed, the temperature of that point is recorded as the solidification point (cooling curve) of the sample. In other words, it is the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase in liquid sample.

A-4.1 Apparatus

A-4.1.1 Thermometers — with graduations of 0.1 and 0.5°C conforming to IS : 2480 (Part 1) - 1983*.

A-4.1.2 Stirrer — of SS wire of 1 mm diameter (see Fig. 3A).

A-4.1.3 Outer Tube — of heat resistant glass 300-mm length and 50-mm diameter (air jacket) (see Fig. 3B).

A-4.1.4 Inner Tube — heat resistant glass, 200-mm length and 35-mm diameter (see Fig. 3B).

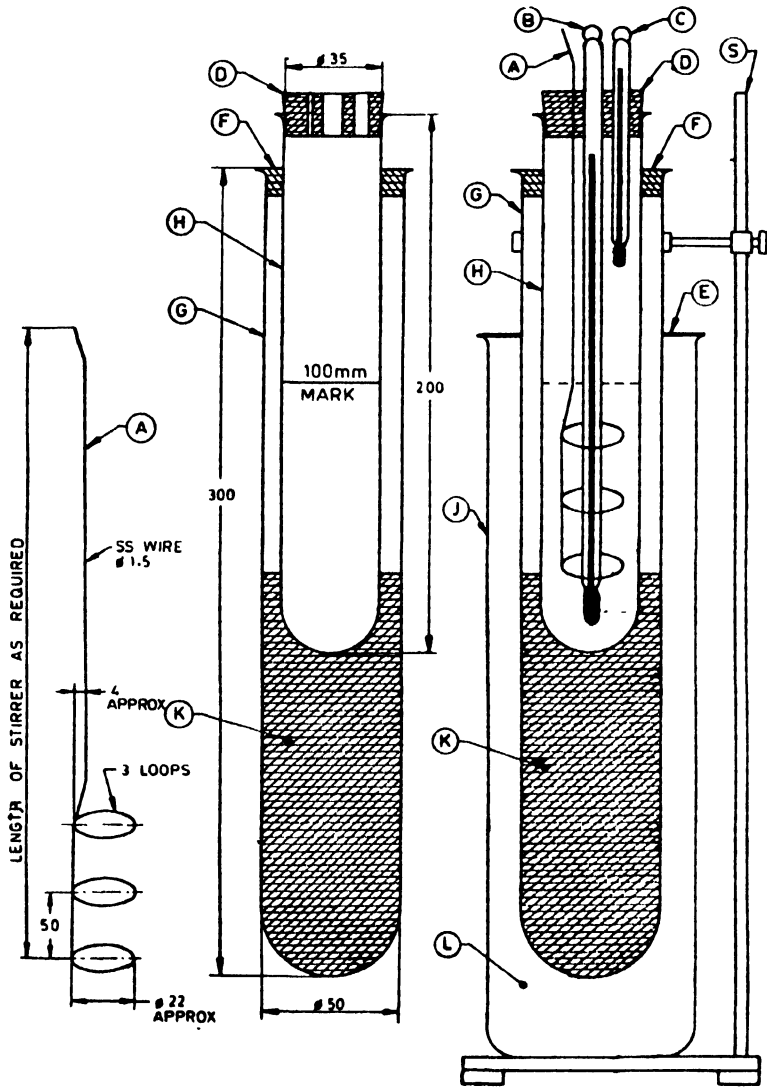
A-4.1.5 Erlenmeyer Flask — 400-ml capacity.

A-4.1.6 Heater

A-4.1.7 Asbestos Board

A-4.1.8 Cooling Bath — suitable container, having effective depth of at least 175 mm; shall be filled with cooling medium (glycerine 60 to 65°C) (see Fig. 3C).

*Specification for general purpose glass thermometers: Part 1 Solid stem thermometers (second revision).



3 A — Stirrers

3 C — Assembly of Apparatus

3 B — Air Jacket and Sampling Tube

A — Stirrer
B — Solidification Point
Thermometer

E — Lid
F — Rubber Cork
G — Air Jacket, Outer Tube
H — Sample Tube, Inner Tube
J — Cooling Bath

K — Cork-Support
L — Glycerine
S — Stand

C — Auxiliary Thermometer
D — Rubber Cork

All dimensions in millimetres.

FIG. 3 APPARATUS FOR SOLIDIFICATION POINT

A-4.2 Reagents

A-4.2.1 Cooling Medium — Technical grade glycerine shall be used as coolant. The temperature of the cooling bath should be kept 5°C less than solidification point of the material.

A-4.3 Preparation of Sample — Take the portion for analysis directly from its container. Melt the entire sample by heating it at about 80°C, that is, 10°C above its solidification point and swirl it for homogeneity before pouring the portion.

A-4.4 Procedure — Hold the cooling bath at 60 to 65°C. Fit the sample container up to its lip into the cork stopper of the air jacket. Fit the thermometer and the stirrer into the two hole stopper of the sample container. Adjust the partial immersion thermometer so that it is immersed up to its 76 mm calibration mark in the sample. The bottom of the thermometer bulb shall then be about 24 mm above the bottom of the sample container.

A-4.4.1 Clamp the assembly to the ring stand and place it so that the air jacket is immersed vertically in the cooling bath to a depth at which at least 100 ml of the length of the sample container is below the surface of the cooling medium.

A-4.4.2 Allow the sample to cool while stirring it at the rate of about 60 strokes per minute. The stirrer should not touch the thermometer or the wall of the sample container. Loop of the stirrer shall not pass through the liquid surface. Discontinue stirring after an appreciable amount of crystals have formed but while the sample is still mainly liquid.

A-4.4.3 Observe and record the thermometer readings at regular intervals, estimating degrees to the nearest 0.1°C until the temperature falls to a minimum, rises to a maximum and remains constant for essential period of time and finally begins to drop. The constant maximum temperature is recorded as the solidification point. As the temperature approaches this point on the rise, thermometer readings should be taken at 10 seconds intervals in order to make sure that the temperature has reached its maximum, and at least three more readings should be taken at maximum level, until the first drop in temperature is noted. Note the experimental maximum temperature, t_e .

NOTE 1 — The accuracy of the thermometer readings can be increased by using a magnifying glass that assures a reading on a line of sight perpendicular to the stem of the thermometer. Also, for the record, the average temperature t_a , of the emergent mercury column should be taken.

NOTE 2 — If the temperature rise after initial crystallization exceeds 0.5°C, remelt the sample by warming it gently in the sample container and repeat the test, seed with two or three small crystals of the sample when the temperature is 0.2 to 0.6°C below the expected solidification point. As an alternative to induce crystallization, a chilled wire may be substituted in place of small crystals of sample.

A-5. DETERMINATION OF COLOUR

A-5.0 Principle—The colour of 50 percent aqueous solution of caprolactam is compared with standard APHA solution. The colour of caprolactam solution is a measure of content of impurities as it varies with the concentration of yellow coloured dissolved impurities.

A-5.1 Apparatus

A-5.1.1 Colourless Nessler Tubes — 100-ml capacity.

A-5.2 Reagents

A-5.2.1 Standard Solution of 5, 10, 15, 20, 30, 40 APHA — One litre solution of 1.245 g of potassium chloroplatinate (K_2PtCl_6), 1.0 g of cobalt chloride ($CoCl_2 \cdot 6H_2O$) and 100 ml of hydrochloric acid (sp gr 1.19) in distilled water corresponds to 500 APHA.

A-5.3 Procedure — Prepare 50 percent aqueous solution of caprolactam and compare visually the colour of the solution with standard APHA solutions (A-5.2.1) in two perfectly colourless and equal Nessler tubes.

A-5.3.1 Report the intensity of colour of the 50 percent aqueous solution in APHA.

A-6. DETERMINATION OF IRON CONTENT

A-6.0 Principle — Ferrous iron, in a dilute hydrochloric acid solution, forms a red-coloured complex with 1-10-phenanthroline. The intensity of the colour is measured at 510 nm by means of a photo-electric spectrophotometer.

A-6.1 Apparatus

A-6.1.1 Photometer — Any photo-electric spectrophotometer or filter photometer that can measure accurately the transmittance of the solution in the range from 500 to 520 nm.

A-6.1.2 Dishes — High silica glass, silica or porcelain, 50-ml and 100-ml capacity.

A-6.1.3 Watch Glasses

A-6.2 Reagents

A-6.2.1 Hydrochloric Acid — Dilute 1 volume of reagent grade HCl (sp gr 1.19) with 19 volumes of water.

A-6.2.2 Standard Iron Solution — Dissolve 0.1000 g of pure iron wire (minimum 99.85 percent iron) in 10 ml of H_2SO_4 (1 : 9) and 3 ml of HNO_3 (sp gr 1.42). Dilute with water to 1 litre in a volumetric flask. One millilitre of this solution contains 0.1 mg of iron.

A-6.2.2.1 Pipette 100 ml of standard iron solution (1 ml = 0.1 mg Fe) into a 1-litre volumetric flask and dilute to the mark with HCl (1:19) (standardize with KMnO_4 for iron content). One milliliter of this solution contains 0.01 mg of iron.

A-6.2.3 Hydroxylamine Hydrochloride Solution — Dissolve 10 g of reagent hydroxylamine hydrochloride in 190 ml of water. This reagent is stable at room temperature.

A-6.2.4 Hydroquinone Solution — Dissolve 2.5 g of hydroquinone reagent or photographic grade, in 100 ml of HCl (1 : 200). Keep in a refrigerator in amber coloured bottle at about 10°C, when not in use.

A-6.2.5 1-10-phenanthroline Solution — Dissolve 0.5 g of 1-10-phenanthroline in 500 ml of water.

A-6.2.6 Sodium Acetate Solution — Dissolve 100 g of reagent grade $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ in 400 ml of water.

A-6.3 Procedure — Weigh 100 ± 0.1 g of the sample into a high-silica glass, silica or porcelain dish. If the iron solution content is less than 5 ppm, use a mass of sample such that the solution on which the spectrophotometer reading is made, contains from 0.005 to 0.050 mg of iron.

Place the dish in an electrically heated muffle furnace and raise the temperature slowly until the sample is completely charred. Then raise the temperature to 500°C and maintain at 500 to 550°C until the sample is greenish white. This usually requires 4 to 6 hours but no harm will be done if the sample is allowed to remain in the muffle overnight. Remove the dish from the muffle furnace, cool to room temperature and add 5 ml of HCl (1 : 1) in such a manner that any ash on the sides of the dish is washed to the bottom. Cover the dish with a watch glass and heat just to boiling.

Transfer the sample to a 100-ml volumetric flask, using water to wash the last trace from the dish and watch glass.

Add 2 ml of hydroxylamine hydrochloride or hydroquinone solution, 5 ml of 1-10-phenanthroline solution and 5 ml of sodium acetate solution, using volumetric pipette. Mix thoroughly after each addition and make to the mark in volumetric flask of 100 ml with water.

Finally, run a blank alongwith the samples to be sure that none of the reagents have become contaminated (*see* Note 2) and for a reference solution against which the transmittance of the sample is measured. Mea-

sure the transmittance of the blank with spectrophotometer, adjust to read 100 percent transmittance at a wave length 510 nm using 5 cm cell, when the absorption cell contains water.

Measure the transmittance of the sample solution at 510 nm using 5.0 cm cell with the spectrophotometer adjusted to read 100 percent for the blank. Read the iron content of the sample solution in milligrams from the calibration curve.

A-6.4 Calculation — Calculate the iron content of the sample in parts per million as follows:

$$\text{Iron, ppm} = \frac{A}{B} \times 1\,000$$

where

A = milligrams of iron found, and

B = quantity of sample in g represented in aliquot used.

A-6.4.1 Check Determination — Make a single determination daily using 1 to 5 ml of standard iron solution (1 ml = 0.01 mg Fe) and proceed as described in A-6.3. If the determined and known values do not agree with the limits of experimental error (± 1 percent transmittance), repeat the test. If the second result do not agree, prepare a new standard solution. If determined and known values do not check after preparing a new standard, investigate for errors in technique and reagents, and if none can be found, prepare a new calibration curve.

A-6.5 Preparation of Calibration Curve

A-6.5.1 Pipette 10, 20, 30, 40 and 50 ml aliquots of standard iron solution into 100-ml measuring flask. Develop colour in the solution as described in A-6.3. Measure the transmittance of the solution at 510 nm using 5 cm cell with the spectrophotometer adjusted to read 100 percent transmittance for the reagent blank. Using semilogarithmic paper, plot the percentage transmittance of the solution against milligrams of iron present.

NOTE 1 — Platinum or platinum-rhodium dishes are not recommended as they sometimes cause a colour interference with the phenanthroline reagent.

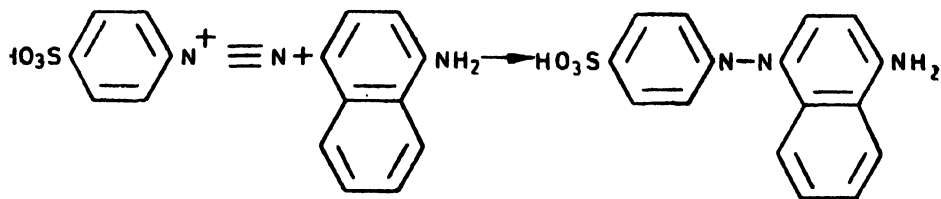
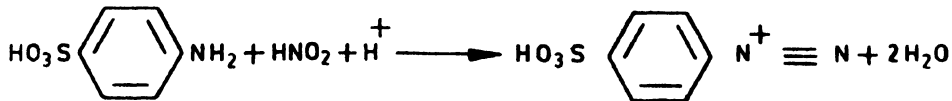
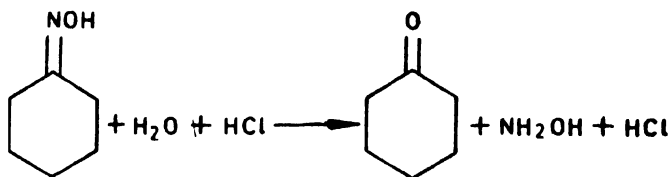
NOTE 2 — When the reagents contain more than 0.002 or 0.003 mg of iron, prepare new reagents and look for the source of contamination.

A-7. DETERMINATION OF OXIME CONTENT

A-7.0 General — This method estimates indirectly, the amount of oxime in caprolactam up to 20 ppm.

A-7.1 Principle — Oxime in caprolactam is hydrolyzed by HCl to cyclohexanone and hydroxylamine. The latter is then oxidized by iodine to an equivalent amount of HNO_2 . The nitrous acid formed is made to react with sulphanilic acid to form diazonium salt. The solution at this stage is then buffered with sodium acetate, followed by removal of excess of iodine with sodium thiosulphate. The diazotized-sulphanilic acid is coupled with 1-naphthylamine, giving scarlet-red (violet) coloured solution. The intensity of the colour is measured using spectrophotometer at 520 nm and 10 nm cell.

A-7.1.1 Reactions



A-7.2 Apparatus

A-7.2.1 Flask — with ground glass stopper and Liebig condenser, capacity 500-ml.

A-7.2.2 Cylinders — graduated, capacity 25-ml and 100-ml.

A-7.2.3 Pipettes — capacity 5-ml, 15-ml and 50-ml.

A-7.2.4 Volumetric Flasks — capacity 100-ml.

A-7.2.5 Funnel — 55 mm dia.

A-7.2.6 Microburette -- capacity 10-ml.

A-7.3 Reagents

A-7.3.1 Hydrochloric Acid — 11 N, 32 percent (1.16 sp gr).

A-7.3.2 Sulphanilic Acid Solution — Take 10.0 g of sulphanilic acid in 750-ml of water and 250 ml glacial acetic acid, and warm the mixture for the complete dissolution and cool to room temperature. Store the solution in dark coloured bottle.

A-7.3.3 Iodine Solution (0.1 N) — Dissolve 12.7 g iodine and 16.0 g KI in water. Make up the volume to one litre and store the solution in a dark bottle.

A-7.3.4 Sodium Acetate — Use special grade sodium acetate ($\text{CH}_3\text{-COONa} \cdot 3\text{H}_2\text{O}$) stored in a dark bottle.

A-7.3.5 1-Naphthylamine Solution — Weigh 3.0 g of the compound and dissolve it in a mixture of 750 ml H_2O and 300 ml of glacial acetic acid. Store the solution in a dark coloured bottle.

A-7.3.6 Sodium Thiosulphate Solution (0.1 N) — Weigh 24.82 g of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) and dissolve in water and make up to one litre.

A-7.3.7 Standard Cyclohexanone Oxime Solution — Weigh accurately 1.0 ± 0.0001 g of cyclohexanone oxime and dissolve it in water and make up to the mark in a one-litre volumetric flask. Concentration of solution is 1 mg/ml. Ten millilitre of this solution is further diluted to one litre. Concentration of the solution is 0.01 mg/ml.

A-7.4 Procedure — Weigh 10.0 g of caprolactam in the 250-ml flask. Add 15 ml hydrochloric acid (32 percent) and 40 ml water. Reflux the mixture with water cooled condenser for one hour. Cool to 20 - 25°C (*see* Note 1), transfer the solution quantitatively to a 100-ml measuring flask and make up the volume and mix. Pipette 50 ml of this solution to a second 100-ml measuring flask. Add 5.0 ml of sulphanilic acid solution and 5.0 ml of iodine solution, shake well and leave aside for exactly 10 minutes (*see* Note 2).

Add 9.5 g of sodium acetate and dissolve it completely, leave aside for exactly 10 minutes (*see* Note 3), then add 5.0 ml of sodium thiosulphate solution and shake well. Immediately there after (*see* Note 4)

add 15.0 ml of 1-naphthylamine solution and make up to mark with water and mix thoroughly. Leave aside for 15 minutes (*see* Note 5), and measure the optical density (light absorption) with photoelectric spectro photometer at 520 nm using 10 mm cell (*see* Note 6) where optical density (light adsorption value) of the blank (*see* Note 7) including all the reagents shall be measured simultaneously for adjustments.

NOTE 1 — Temperature of the liquid in the case of oxidation with iodine influence the result. Care should, therefore, be taken to avoid a big difference in temperature from that at which the calibration curve is prepared.

NOTE 2 — Time should be exactly 10 minutes as light absorption decreases with lapse of time.

NOTE 3 — Time should be exactly 10 minutes to obtain the right light absorption. The light absorption value is influenced by the time elapsed after sodium acetate is added.

NOTE 4 — 1-Naphthylamine solution should be added immediately. The light absorption value is influenced by the time elapsed after sodium thiosulphate is added.

NOTE 5 — The light absorption value continues to increase rapidly during the first 10 minutes after addition of 1-naphthylamine. Then the increase rate becomes lower. The tolerance would, therefore, be 15 ± 2 minutes.

NOTE 6 — Distilled water should be used for the control solution for light absorption determination.

NOTE 7 — Strictly speaking, pH of the blanks should be the same as that of the sample. Practically, however, any pH adjustment is not required because pH difference usually do not cause much difference in the results.

A-7.5 Preparation of Calibration Curve — Transfer 0, 3.0, 5.0, 10.0, 15.0 and 20.0 ml of standard oxime solution (0.01 mg/ml) in different 250-ml conical flasks (add to each of them 10 g of lactam with a small amount of cyclohexanone oxime) and then add 40-ml of water. Then follow the details as given in procedure (*see* A-7.4), and determine the net optical density by deducting the blank from observed optical density.

A-7.6 Calculation

$$\text{Cyclohexanone oxime content} = \frac{2 \times M_2 \times 1\,000}{M_1}$$

where

M_2 = milligram read from the graph, and

M_1 = mass in mg of the sample taken.

A-7.7 Report — Report the amount of cyclohexanone oxime present in caprolactam sample as the mg/kg (ppm).

A-8. DETERMINATION OF FREE ACIDITY OR ALKALINITY

A-8.0 General — The acidity/alkalinity of caprolactam is determined by titration using Tashiro indicator.

A-8.1 Apparatus

A-8.1.1 Erlenmeyer Flask with Stopper — 250-ml capacity.

A-8.1.2 Burette — 25-ml capacity with graduation interval of 0.05 ml.

A-8.1.3 Analytical Balance

A-8.2 Reagents

A-8.2.1 Sodium Hydroxide Solution — 0.01 N.

A-8.2.2 Sulphuric Acid — 0.01 N.

A-8.2.3 Tashiro Indicator — Measure 28 ml of methyl red (0.1 percent by mass in distilled water) in a 100-ml measuring flask and add 7 ml of methylene blue (0.1 percent by mass in distilled water) and make up to the mark.

A-8.3 Procedure — Take 50-ml of distilled water in Erlenmeyer flask and neutralize it using one drop of Tashiro indicator. Weigh 50 g of caprolactam into the Erlenmeyer flask and shake to dissolve it. Titrate the solution with 0.01 N sodium hydroxide or 0.01 N sulphuric acid as required using one drop of Tashiro indicator.

A-8.4 Calculation

$$\text{Acidity/alkalinity, meq/kg} = \frac{V \times N}{M} \times 1000$$

where

V = volume of NaOH or H_2SO_4 used for titration of sample in ml,

N = normality of NaOH or H_2SO_4 , and

M = mass in g of sample taken for test.

NOTE — If the solution of caprolactam turns the neutral solution of Tashiro indicator red, express the result as acidity. If the neutral solution turns green, express the result as alkalinity.

A-9. DETERMINATION OF MOISTURE CONTENT

A-9.0 Principle — The moisture in caprolactam is determined by titrating it with standard Karl Fischer Reagent to an electrometric end point.

A-9.1 Apparatus

A-9.1.1 Burette — 10-ml burette graduated in 0.05 ml, fitted with three-way stopcock.

A-9.1.2 Electrodes — Platinum wire approximately 25 mm long and 0.5 mm diameter.

A-9.1.3 Electric Circuit — A dc micrometer of 0 to 50 μ A range and internal resistance approximately 1 500 ohms.

A-9.1.4 Potentiometer with a Resistance of 2 000 ohms

A-9.2 Reagents

A-9.2.1 Pyridine

A-9.2.2 Oxalic Acid — pure.

A-9.2.3 Sodium Tartrate Dihydrate

A-9.2.4 Gas Cylinder of SO₂

A-9.2.5 Iodine — sublimed.

A-9.2.6 Methanol — Anhydrous, containing less than 0.1 percent of water.

A-9.2.7 Concentrated Sulphuric Acid — sp gr 1.84.

A-9.2.8 Karl Fischer Reagent, Stock Solution — Dissolve 85 ± 1 g of iodine in 270 ± 2 ml of pyridine in a dry glass-stoppered bottle. Add 670 ± 2 ml of methanol (see A-9.2.6). Cool the mixture in an ice bath to below 3.9 °C. Bubble gaseous sulphur dioxide, dried through concentrated sulphuric acid, into the cooled mixture to achieve an increase in mass by 65 g.

A-9.2.9 Karl Fischer Reagent Standard Solution — Add 50 ml of the methanol to a clean, dry titration flask. Insert the stopper and adjust the magnetic stirrer to give a smooth stirring action. Turn on the indicating circuit and add Karl Fischer reagent in suitable amounts. At first, the magic eye will remain open due to local concentration of unreacted reagent around the electrodes but will close as the reagent is consumed. As the end point is approached, the magic eye will close down slowly, after each addition of Karl Fischer reagent at the end point, the eye will remain open at least for 30 seconds.

Add 1 drop of distilled water to the solution in the titration flask from a weighing pipette previously weighed to the nearest 0.1 mg. Stopper the flask. Re-weigh the weighing pipette. Titrate to the end point.

A-9.2.9.1 Calculate the water equivalence of the Karl Fischer reagent as follows:

$$F = \frac{W}{T}$$

where

F = water equivalence of Karl Fischer reagent in mg/ml;

W = water added, mg; and

T = quantity of reagent required for titration of water added, ml.

A-9.3 Procedure -- Assemble the apparatus as shown in Fig. 4. Add 50-ml of methanol to the titration flask and titrate with standard Karl Fischer reagent to the end point. Stopper the sample inlet tube as quickly as possible to prevent absorption of moisture from the atmosphere.

Weigh accurately about 10 g of the sample and add to the titration flask. Titrate the sample to the end point. Record the quantity of reagent used.

A-9.4 Calculation

$$\text{Moisture, ppm} = \frac{CF \times 1000}{M}$$

where

C = quantity of reagent required for titration of the sample, ml;

F = water equivalence in ml of water per ml; and

M = mass of sample taken, g.

A-10. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-10.0 Principle — A weighed quantity of caprolactam is dissolved in distilled water. The solution is filtered and washed through tared sintered glass crucible (G_4). The crucible is dried and weighed again. The difference in two mass is the matter insoluble in water present in the sample taken.

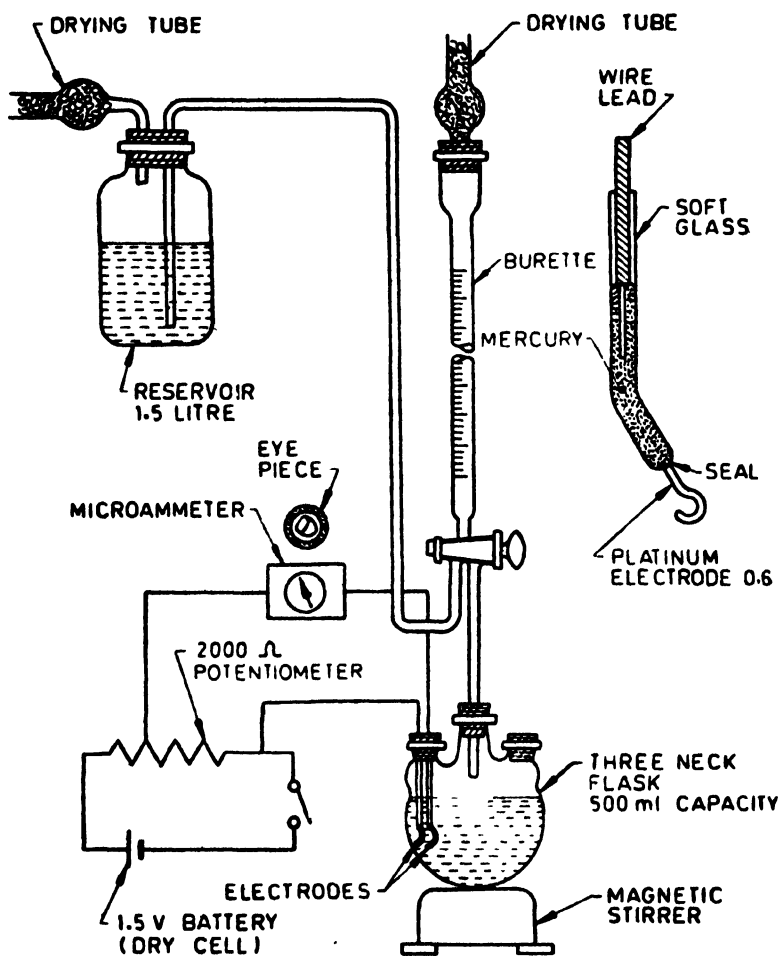


FIG. 4 APPARATUS FOR DETERMINATION OF WATER BY KARL FISCHER REAGENT

A-10.1 Apparatus

A-10.1.1 Sintered Glass Crucible (G_4) — 30-ml capacity.

A-10.1.2 Wash Bottle

A-10.1.3 Beaker — 2.5-litre capacity.

A-10.1.4 Oven

A-10.1.5 Desiccator

A-10.2 Reagents

A-10.2.1 Acetone

A-10.2.2 Double Distilled Water

A-10.3 Procedure

A-10.3.1 Wash the sintered glass crucible (G_4) with 200 ml of double distilled water followed by 100 ml of acetone. Dry the crucible at 110 °C in a drying oven for at least 15 minutes. Place the crucible in a desiccator, allow it to cool for 45 minutes and weigh accurately to four places of decimal (M_0).

A-10.3.2 Weigh $1\,000 \pm 1$ g of caprolactam into a 2.5-litre beaker, add 1 000 ml of double distilled water and stir. Warm it, if necessary, to promote dissolving. Filter this solution through the crucible placed on a suction flask and wash the beaker with water. Wash crucible with 200 ml of distilled water.

Dry the crucible at 100°C in a drying oven for at least 30 minutes. Place the crucible in a desiccator, allow it to cool for 45 minutes and weigh again to four decimal places (M_1).

A-10.4 Calculation

A-10.4.1 Matter insoluble in water, ppm =
$$\frac{(M_1 - M_0) \times 10^6}{M}$$

where

M_1 = mass in g of crucible and residue,

M_0 = mass in g of empty crucible, and

M = mass in g of sample taken.

A-10.4.2 Report — Report the results in ppm of matter insoluble in water which is present in the sample.

A-11. DETERMINATION OF ABSORBANCE AT A WAVELENGTH OF 290 nm (SPECTROPHOTOMETRIC METHOD)

A-11.0 General — Absorbance of aqueous solution of caprolactam is a measure of the degree of its purity.

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A-11.1 Principle — The absorbance of 50 percent aqueous solution of caprolactam is spectrophotometrically determined in 1 cm quartz cell at 290 nm.

A-11.2 Apparatus

A-11.2.1 Ultraviolet Molecular Absorption Spectrometer — fitted with a hydrogen or deuterium lamp.

A-11.2.2 Quartz Cells — two, optical path length, 1 cm.

A-11.3 Procedure

A-11.3.1 Spectrometric Measurements — Weigh accurately 50.0 ± 0.1 g of the sample, dissolve in 50 ml of distilled water and mix well. Filter the solution through Whatman filter paper No. 40 or equivalent to remove any undissolved foreign material. Bring the solution to room temperature and let it stand until all the air bubbles disappear. Fill one of the quartz cells with the test solution and fill the other cell with water. Carry out the spectrometric measurements at 290 nm, after having adjusted the instrument to zero absorbance against water.

A-11.3.2 Correction for Absorbance of Cells — Fill the two cells used for the measurements (A-11.3.1) with distilled water and measure the absorbance of each cells at a wavelength of 290 nm. Let one of the cells read 'zero' since it has been used to adjust the instrument to zero absorbance.

NOTE — The difference between the measured absorbances should not exceed 0.003.

A-11.4 Calculation — Calculate the absorbance of 50 percent aqueous solution of caprolactam as under:

$$\text{Absorbance of caprolactam (50 percent aqueous solution) in 1 cm quartz cell at 290 nm} = \frac{A_1 - A_0}{l}$$

where

A_1 = absorbance of the test solution,

A_0 = correction for the differences in absorbance of the cells, and

l = optical path length of the cell, cm.

APPENDIX B

(Clause 4.1)

SAMPLING OF CAPROLACTAM

B-1. SCALE OF SAMPLING

B-1.1 Lot — All the packages in a single consignment of the same size and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

B-1.2 For ascertaining the conformity of the material to the requirement of this specification, samples shall be tested from each lot separately.

B-1.3 The number of packages to be selected from the lot shall depend on the size of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

NUMBER OF THE PACKAGES IN THE LOT	SAMPLE SIZE
(1)	(2)
Up to 25	2
26 to 50	3
51 to 100	5
101 to 300	7
301 and above	10

B-1.3.1 These packages shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-2. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLES

B-2.1 From each of the packages selected according to B-1.3 draw small portions of the material from different parts of the packages. The total quantity of the material drawn from each package shall be sufficient to make triplicate determinations for all the requirements given in the specification.

B-2.2 Mix thoroughly all portions of the material drawn from different parts of each selected package. Out of these portions, a small but approxi-

*Methods for random sampling.

mately equal quantity of material shall be taken and thoroughly mixed so as to form a composite sample sufficient to make triplicate determinations for all the requirements to be tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee. These parts shall be immediately transferred to clean and dry containers which shall then be sealed air-tight and labelled with full details of sampling such as name of the product, name of the manufacturer, batch number and any other particulars of the consignment.

B-2.3 The remaining portions of the material from each package (after a small quantity needed for composite sample has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred to clean and dry containers which are then sealed air-tight and labelled with all the particulars of sampling given in **B-2.2**. The material in each such sealed container shall constitute an individual test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each container selected. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

B-2.4 Referee Samples — Referee samples consist of the composite sample (*see B-2.2*) and a set of individual sample (*see B-2.3*) marked for this purpose and shall be kept at a place agreed to between the two so as to be used in case of a dispute.

B-3. NUMBER OF TESTS

B-3.1 Permanganate number, solidification point, colour and absorbance at 290 nm shall be tested on each of the individual samples.

B-3.2 The remaining requirement given in 2 shall be tested on the composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For each of the requirements (**B-3.1**) tested on individual samples, all the test results shall meet the corresponding requirement in Table 1.

B-4.2 All the test results on the composite sample shall meet the corresponding requirement in Table 1.

B-4.3 The lot shall be declared as conforming to the requirements of the specification if **B-4.1** and **B-4.2** are satisfied.

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